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FORM I		90 (Modified)  U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE  ATTORNEY'S DOCKET NUMBER						
	Th	RANSMITTAL LETTER TO THE UNITED STATES 4296						
		DESIGNATED/ELECTED OFFICE (DO/EO/US)  U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR						
		CONCERNING A FILING UNDER 35 U.S.C. 371 09/646978						
INTE PC'	RNAT T/J	rional application no. international filing date PRIORITY date Claimed January 27, 2000 January 28, 1999						
_		INVENTION						
L.	IGH	T-EMITTING MATERIAL AND PRODUCING METHOD THEREOF						
APPL	JCAN'	T(S)FOR DO/EO/US 1) Qinglong HAO, 2) Pengcheng LI, 3) Qian XIU,						
		4) Atsushi OGURA and 5) Jingfeng GAO						
Appli	icant l	herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.						
I.	X	This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.						
2.		This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.						
3.		This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).						
4.		A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.						
5.	X	A copy of the International Application as filed (35 U.S.C. 371 (c) (2))						
		a.  is transmitted herewith (required only if not transmitted by the International Bureau).						
		b. 🛮 has been transmitted by the International Bureau.						
		c. $\square$ is not required, as the application was filed in the United States Receiving Office (RO/US).						
<u>4</u> 6.	X	A translation of the International Application into English (35 U.S.C. 371(c)(2)).						
<b>7</b> .		A copy of the International Search Report (PCT/ISA/210).						
<b>≈</b> .8.		Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))						
i.		a.   are transmitted herewith (required only if not transmitted by the International Bureau).						
Took state from the law took		b.  have been transmitted by the International Bureau.						
iii		c. have not been made; however, the time limit for making such amendments has NOT expired.						
<b>3</b> 1:		d.  have not been made and will not be made.						
<b>=</b> 9.		A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).						
=9. <b>T</b> 0.	X	An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).						
LI.	1.   A copy of the International Preliminary Examination Report (PCT/IPEA/409).							
<b>12</b> .		A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).						
In	tems 1	13 to 18 below concern document(s) or information included:						
13.		An Information Disclosure Statement under 37 CFR 1.97 and 1.98.						
14.		An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.						
15.	<u></u>	A FIRST preliminary amendment.						
-	•	A SECOND or SUBSEQUENT preliminary amendment.						
16.	. 🗆	A substitute specification.						
17.		A change of power of attorney and/or address letter.						
18.	<u></u>	Certificate of Mailing by Express Mail						
19.	<b>S</b>	Other items or information:						
		THE FEE CACULATION MUST BE BASED ON THE CLAIMS AS AMENDED AND ADDED IN THE ATTACHED PRELIMINARY AMENDMENT.						

# 529 Rec'd PCT/PTO 25 SEP 2000

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The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. 01-1944 A duplicate copy of this sheet is enclosed.											
NOTE: 1.137(a)	NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.										
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### 529 Rec'd PCT/PTO 25 SEP 2000

#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE MATTER OF:

APPLICANT: HAO et al

ORDER/DOCKET NO. 4296

FOR: LIGHT-EMITTING MATERIAL AND PRODUCING METHOD THEREOF

### PRELIMINARY AMENDMENT

Hon. Commissioner of Patents and Trademarks Washington, DC 20231

SIR:

Please amend the application as follows.

#### IN THE CLAIMS

Please amend claim 8 as folllows:

Claim 8, line 2, delete "or 7"

Please add new claim 9 as follows:

- - 9. A producing method of a light-emitting material according to claim 7, wherein in said step (2), reduction is carried out using carbon powder. - -

#### **REMARKS**

A Preliminary Amendment has been made to correct multiple dependent claim 8.

It is respectfully requested that the above amendment be entered before calculation of the filing fee and before examination by the Examiner.

Respectfully submitted,

Eugene Lieberstein Registration No. 24645

ga 0721 ha. amd

Anderson, Kill & Olick, P.C. 1251 Avenue of the Americas New York, NY 10020-1182 212/278-1000

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#### **SPECIFICATION**

LIGHT-EMITTING MATERIAL AND PRODUCING METHOD THEREOF

#### TECHNICAL FIELD

The present invention relates to a light-emitting material and a producing method thereof, and more particularly, to an inorganic light-emitting material using a rare-earth element as an exciting agent and having an afterglow time and a producing method thereof.

#### BACKGROUND TECHNIQUE

A light-emitting material is utilized is mixed in ink or paint to make light emitting paint, and is utilized on a safe sign and a clock board. Conventionally, copper-excited zinc sulfide (ZnS: Cu) was widely used as the light-emitting material. ZnS: Cu has high light-emitting efficiency in the light-emitting spectral region, but its brightness is lowered extremely rapidly, and the visible afterglow time is as short as 20 to 30 minutes. When the ZnS: Cu is exposed to ultraviolet rays in areas exposed to moisture, decomposition and degeneration are generated and the body color of the material darkens. Therefore, there are constraints for using ZnS: Cu outside, and optimal material as a substitute for ZnS: Cu had long been required.

In CN1053807A, a light-emitting material (m(Sr<sub>1-x</sub>Eu) O·nAl<sub>2</sub>O<sub>3</sub>-yB<sub>2</sub>O<sub>3</sub>) having long afterglow ability and is laid open. In the above general formula,  $1 \le m \le 5$ ,  $1 \le n \le 8$ ,  $0.005 \le y \le 0.35$  and  $0.001 \le x \le 0.1$ . The afterglow time of this light-emitting material is in a range from 10 to 20 hours.

In USP5,376,303, phosphor having long afterglow ability comprises a compound (MO a (Al<sub>1-b</sub>B<sub>b</sub>)  $_2O_3$ : cR).

In this general formula,  $0.5 \le a \le 10.0$ ,  $0.0001 \le b \le 0.5$ ,  $0.0001 \le c \le 0.2$ , MO is a at least one compound selected from a group consisting of MgO, CaO, SrO and ZuO. R consists Eu and at least one additive rare-earth element selected from a

group consisting of Pr, Nd, Dy and Tm.

In the above-mentioned patent, some phosphors such as SrO. 2.10  $(A_{10.952}B_{0.048})$   $_2O_3$ : 0.005Eu, 0.020Dy (which will be referred to as "A" hereinafter), and  $SrO._{1.025}$   $(Al_{0.976}B_{0.024})_2O_3$ : 0.005Eu, 0.015 Dy (which will be referred to as "B" hereinafter) were prepared, and residual light-emitting time and brightness of these phosphors were evaluated in view of afterglow time constant (n) and relative brightness (ZnS: Cu, Cl were evaluated). A measuring result shows that the afterglow time constants of A and B phosphors and ZnS: Cu, Cl are 0.94, 0.86 and 1.26, respectively, and phosphorus brightness after 10 seconds are 144, 220 and 100, respectively, and phosphorus brightness after 20 seconds are 934, 1320 and 100, respectively.

It is found that these materials are clearly improved as compared with ZnS: Cu, Cl, but they are not yet in practical

Thereupon, the present inventors conducted various researches for producing light-emitting material using rare-earth element Eu as the light-emitting material. Based on the researches, the present inventors found that a light-emitting material having a new crystallization structure was obtained by adding an appropriate amount of B and an additive exciting agent. This light-emitting material has a desired long afterglow time and high brightness.

Therefore, a first object of the present invention is to provide a light-emitting material having a long afterglow time and high brightness.

Moreover, a second object of the present invention is to provide a producing method of the above-mentioned light-emitting material.

#### DISCLOSURE OF THE INVENTION

To achieve the objects, the present invention provides the following light-emitting material and the producing method. That is, a light-emitting material of the present invention includes a diplophase compound crystallization expressed in a general formula:  $(Sr, Eu, Dy)_{0.95\pm x}(Al, B)_{2}O_{3.95}$   $_{\pm x}$   $\cdot$   $(Sr, Eu, Dy)_{4-x}(Al, B)_{14}O_{25-x}(x=0.01 \sim 0.1)$ .

This diplophase compound has a new crystallization structure and consists of two phases, i.e., (Sr, Eu, Dy)  $_{0.95}$   $_{\pm x}$  (Al, B)  $_2O_3._{95\pm x}$  and (Sr, Eu, Dy)  $_{4-x}$ (Al, B)  $_{14}O_{25-x}$ . This conclusion was obtained from XRD (X-ray diffraction) analysis using a Chinese geological university material scientific divisional crystallization structure and a large amount of samples carried out in a crystallization chemical laboratory. Based on the XRD analysis, and using an optical microscope and electronic probe analytical technique, it is corroborated that the above two phases are symbiosis and have light-emitting function.

The producing method of the light-emitting material includes the following steps:

- (1) step for measuring previously pulverized raw materials, and mixing them to obtain a mixture of raw material,
- (2) step for putting the mixture into a container, heating the mixture from  $850^{\circ}$ C to  $1200^{\circ}$ C for three hours under a reduction condition, keeping the temperature for five to six hours, thereby obtaining a sintered body,
- (3) step for stopping the heating operation and cooling the sintered body nature down to a room temperature, and
- (4) step for pulverizing the sintered body to obtain a product.

According to the light-emitting material and the producing method of the present invention, there is a light-emitting effect that which visible long afterglow ability as compared with a similar light-emitting material.

#### BEST MODE FOR CARRYING OUT THE INVENTION

In an assessment process concerning a phase of a light-emitting material of the present invention, using analytic means such as an X-ray fluorescent analysis, a plasma spectral analysis, electronic probe and X-ray photoelectron

energy spectrum, it was confirmed that a content of B element in diplophase compound is  $0.2\sim1.0$  % by weight in general, and variable ranges of contents of Eu element and Dy are from 0.5 to 3.0 % by weight and from 0.01 to 3.0 % by weight, respectively.

The element B exists in the entire crystallization structure. Moreover, the element B exists in a form of B-O tetrahedral coordination or BO<sub>3</sub> triangular coordination. The BO<sub>3</sub> Triangular coordination can substitute a portion of Al-O octahedron and this causes instability in the crystallization structure. This is an important structural feature of the light-emitting material of the present invention.

Further, the Al-O octahedron and Al-O tetrahedron concurrently exist in the diplophase compound crystallization of the present invention, and form a substantially hexagonal ring and positive ions of Sr, Eu and Dy are charged into a cavity of the ring. From the viewpoint of the entire crystallization diplophase compound, Al exists excessively and (Sr, Eu, Dy) are insufficient.

The raw materials which is used for the producing method of the light-emitting material of the present invention are  $SrCO_3$ ,  $Al_2O_3$ ,  $H_3BO_3$ ,  $Eu_2O_3$  and  $Dy_2O_3$ , of which, Eu3+ of  $Eu_2O_3$  is reduced by Eu2+ during sintering process to excite the diplophase compound and provide the same with a light-emitting function.  $Dy_2O_3$  strengthens the exciting effect of  $Eu_2O_3$  as an additive exciting agent.

The term "reduction condition" used in the present invention means to reduce the above-mentioned mixed raw material using carbon powder, or to reduce the mixed raw material using mixture gas of nitrogen and hydrogen of volume ratio of 4:1. The light-emitting material produced by the invention has faint yellow-green color. When this light-emitting material is irradiated with sunlight, a fluorescent light or the other artificial light source and excited, the main peak of the light-emitting spectrum is  $505\,\mu\text{m}$ , and shows

blue or green.

As a result of measurement of samples, it was found that the light-emitting material of the present invention showed brightness of about 8500mcd/m² after five seconds from the instant when the irradiation was stopped, and visible afterglow time was 80 hours or longer (see Table 1). As shown in Table 1, the light-emitting material produced by the method of the present invention has especially excellent visible afterglow time.

The brightness is measured by the following method. That is, 0.2g of sample is put in a plastic plate of 10mm diameter and it is irradiated for 15 minutes from a perpendicular distance of 20cm using a fluorescent light of 15w at a room temperature and under humidity of 25RH%, and brightness of each sample is measured at various time points using an luminance meter (TOPCONBM-5, Japan TOPCON Inc.).

The light-emitting material produced by the method of the present invention has apparently long afterglow time in comparison with similar other products. Therefore, this material can suitably be applied to articles or safe sign which need to be seen in the dark, for example, a fireplug of a fire extinguishing tools and material, a handrail of safe stairs, and a road.

The following embodiments are for explaining the present invention in more detail, and are for limiting the invention. [First Embodiment]

Previously pulverized 372.89g of  $SrCO_3$ , 220.32g of  $Al_2O_3$ , 12.616g of  $H_3BO_3$ , 2.42g of  $Eu_2O_3$ , and 0.157g of  $Dy_2O_3$  were measured and sufficiently mixed. The mixed raw material was put into a container and it was covered with carbon powder, and heated from  $850^{\circ}$ C to  $1200^{\circ}$ C for three hours to increase it temperature, and the temperature was maintained for six hours. Then, the mixture was naturally cooled down to a room temperature to obtain a sintered body. The obtained sintered body was pulverized into such small pieces that all the pieces could pass through 200 mesh, thereby obtaining a product.

The product obtained in this manner had initial brightness of  $3850\text{mcd/m}^2$  for 30 seconds and afterglow time was 85 hours. In the obtained produce, a value of x in the general formula was 0.01.

#### [Second Embodiment]

Previously pulverized 409.79g of  $SrCO_3$ , 220.32g of  $Al_2O_3$ , 12.616g of  $H_3BO_3$ , 2.96g of  $Eu_2O_3$ , and 0.164g of  $Dy_2O_3$  were measured and sufficiently mixed. The mixed raw material was put into a container and it was covered with carbon powder, and heated from  $850^{\circ}C$  to  $1000^{\circ}C$  for three hours to increase it temperature, and the temperature was maintained for six hours. Then, the mixture was naturally cooled down to a room temperature to obtain a sintered body. The obtained sintered body was pulverized into such small pieces that all the pieces could pass through 200 mesh, thereby obtaining a product.

The product obtained in this manner had initial brightness of  $3990 \text{mcd/m}^2$  for 30 seconds and afterglow time was 80 hours.

In the obtained produce, a value of x in the general formula was 0.01.

Table 1

Measurement of brightness  $(mcd/m^2)$  and calculation of standard deviation

		Sample	No.			Calculation	n of standard deviation	deviation
Time	Н	2	е	4	2	Average value		Relative
							deviation	standard
								deviation
5 S	8400	8450	8400	8500	8500	8430	45	0.5%
10 s	7380	7570	7420	7410	7320	7460	125	1.8%
20 s	5120	5130	4930	5170	4870	5044	135	2.78
30 s	4030	3960	3850	3990	3820	3930	91	2.3%
40 s	3380	3230	3160	3310	3110	3238	110	3.4%
e0 s	2550	2490	2430	2490	2420	2476	53	2.1%
90 s	1870	1820	1780	1830	1750	1810	4.7	2.6%
3min	1520	1470	1460	1480	1420	1470	36	2.4%
4 min	870	850	840	840	820	844	18	2.1%
5 min	760	740	720	730	700	730	22	3.0%
3 min	630	630	610	610	590	614	17	2.8%
15 min	330	310	300	310	290	308	15	4.9%
30 min	160	150	140	140	130	144	15	8.3%
60 min	70	70	09	09	09	64	5.5	8.6%
90 min	50	40	40	20	40	44	5.5	12.5%
120 min	40	40	30	40	30	36	5.5	15.3%
180 min	20	30	20	20	20	22	4.5	20.5%
240 min	20	20	10	20	20	18	4.5	25.0%
360 min	20	10	10	20	10	14	5.5	33.6%
480 min	10	10	10	10	10	10		

#### WHAT IS CLAIMED IS:

- 1. A light-emitting material including diplophase compound that is expressed in the following general formula:
- (Sr, Eu, Dy)  $_{0.95\pm x}$  (Al, B)  $_2O_{3.95\pm x}$  (Sr, Eu, Dy)  $_{4-x}$  (Al, B)  $_{14}O_{25-x}$  (in the formula, x=0.01 to 0.1, a content of B element is 0.2 to 1.0 % by weight, a content of Eu is 0.5 to 3.0 % by weight and a content of Dy is 0.1 to 3.0 % by weight).
- 2. A light-emitting material according to claim 1, wherein said diplophase compound comprises symbiotical phase (Sr, Eu, Dy)  $_{0.95~\pm x}$  (Al, B)  $_{2}O_{3.95\pm}$  from (Sr, Eu, Dy)  $_{4-x}$  (Al, B)  $_{14}O_{25-x}$ .
- 3. A light-emitting material according to claim 1, wherein Al-O tetrahedron and Al-O octahedron concurrently exist in said diplophase compound.
- 4. A light-emitting material according to claim 1, wherein  $BO_3$  triangular arrangement substitute a part of Al-O octahedron in said diplophase compound.
- 5. A light-emitting material according to claim 1, wherein boron exists entirely in said diplophase compound crystalline.
- A producing method of a light-emitting material of claim1, comprising
- step for measuring previously pulverized raw materials,
   and mixing them to obtain a mixture of raw material,
- (2) step for putting the mixture into a container, heating the mixture from  $850^{\circ}$ C to  $1200^{\circ}$ C for three hours under a reduction condition, keeping the temperature for five to six hours, thereby obtaining a sintered body,
- (3) step for stopping the heating operation and cooling the sintered body nature down to a room temperature, and
- (4) step for pulverizing the sintered body to obtain a product.
- 7. A producing method of a light-emitting material according to claim 6, wherein said previously pulverized raw materials are  $SrCO_3$ ,  $Al_2O_3$ ,  $H_3BO_3$ ,  $Eu_2O_3$  and  $Dy_2O_3$ .
- 8. A producing method of a light-emitting material according to claim 6 or 7, wherein in said step (2), reduction is carried out using carbon powder.

#### ABSTRACT OF THE DISCLOSURE

A light-emitting material of the present invention includes diplophase compound that is expressed in the following general formula: (Sr, Eu, Dy)  $_{0.95\pm x}$  (Al, B)  $_{203.95\pm x}$  (Sr, Eu, Dy)  $_{\text{4-x}}$  (Al, B)  $_{14}\text{O}_{\text{25-x}}$  (in the formula, x=0.01 to 0.1, a content of B element is 0.2 to 1.0 % by weight, a content of Eu is 0.5  $\,$ to 3.0 % by weight and a content of Dy is 0.1 to 3.0 % by weight). A producing method of a light-emitting material of the present invention comprises (1) step for measuring previously pulverized raw materials, and mixing them to obtain a mixture of raw material, (2) step for putting the mixture into a container, heating the mixture from 850% to 1200% for three hours under a reduction condition, keeping the temperature for five to six hours, thereby obtaining a sintered body, (3) step for stopping the heating operation and cooling the sintered body nature down to a room temperature, and (4) step for pulverizing the sintered body to obtain a product. In the step (2), reduction is carried out using carbon powder.

## COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER

4296

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

the specification of which (check only one item below):

is attached hereto.

is attached hereto.

on

and was amended

on

was filed as PCT international application

Number

on

and was amended under PCT Article 19

on

(if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowlege the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

#### PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)		Y CLAIMED 15 USC 119
China	99 1 00285.7	January 28, 1999	☑ YES	□ NO
			YES	□ NO
			YES	□ NO
	·		YES	□ NC
			☐ YES	□ NO

C)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowlege the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

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I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowled the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

	PRIOR 35 U.S	U.S. APPLICATIONS OR .C. 120:	PCT INTERNA	TIONAL APPLICATIONS DE	SIGNATING	THE U.S. F	OR BENEFIT	UNDER	
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